Axys Analytical Services Ltd
Standard Operating Procedure

Title: Sample Control Procedure
Area: Laboratory Procedures

Purpose:

To maintain control over samples with respect to (1) sample location; (2) status of each sample (i.e., consumed, disposed of, etc.); (3) the requested analyses; and (4) which analyses have been completed on a particular sample.

Scope:

All samples submitted for analysis are subject to sample control procedures such that the location of a sample and its extracts are always known.

Procedure:

When samples are not being analyzed they are stored according to sample storage requirements. The exact location of a sample is recorded in the Laboratory Information Management System (LIMS). A summary of all storage locations is presented in Table 1. When a sample is removed from its storage location, the following sample control measures must be used.

The Laboratory Services Group is responsible for retrieving and returning samples from the storage area.

1. To request samples, an Extraction Laboratory Chemist completes the Sample Retrieval Log (FSA-017) located in the Laboratory Services area. The request must be made by 3:00 p.m. of the day before the samples are needed. The analyst records the Sample IDs, the date required, and their initials.

2. At the end of each day a Laboratory Services Group Chemist retrieves the requested samples from storage. The Laboratory Services Chemist consults the LIMS or Homogenizing Records located in the Laboratory Services Manager's office to locate the sample. The Laboratory Services Chemist records the original sample location and the date and time the samples were retrieved on the Sample Retrieval Log, and initials the box with the sample IDs.

3. The sample containers are checked for cracks or other damage. If necessary, samples are

transferred to new, clean jars. Sample labels are also transferred to the new jar.

4. The retrieved samples are treated as follows:
   - large jars containing large amounts of frozen, wet, sediment or soil are wrapped in foil and allowed to thaw on the bench-top overnight;
   - smaller samples, or dry sediment and soil samples are placed in the Lab 1 fridge to thaw overnight;
   - tissue samples are placed in the Lab 1 fridge to thaw overnight;
   - jars of XAD resin are placed in the Lab 1 fridge;
   - dry pulp is placed in the Lab 6 cupboard;
   - wet pulp samples are placed in the Lab 1 fridge;
   - glass filter cartridges are placed in the Lab 1 fridge.

   The Laboratory Services Chemist and the Extraction Laboratory Chemist must ensure that frozen samples are not removed from the storage freezers for longer than 24 hours.

5. After subsampling each sample, the Extraction Laboratory Chemist initials and dates the sample label.

6. If there is insufficient sample available for analysis, the Extraction Laboratory Chemist alerts their Manager. The Manager must be consulted before consuming a sample.

7. If any of the samples were completely consumed in the subsampling procedure or if any of the samples were altered in any way, the Extraction Laboratory Chemist makes a note on the Sample Retrieval Log. Consumed sample containers are given to the Lab Services Technician. The Lab Services Technician removes and stores the Labels in the Consumed Samples Binder. All consumed sample IDs are recorded in the Consumed Samples spreadsheet on the network drive.

8. After subsampling all of the samples, the Extraction Laboratory Chemist returns the samples to the fridge in Lab 1. The Extraction Laboratory Chemist initials the "Sample Return Request" box on the Sample Retrieval Log.

9. At the end of each day, it is the responsibility of a Laboratory Services Chemist to return

samples to their original storage location. This activity may be initiated by a request from an Extraction Laboratory Chemist. As noted above, frozen samples are not removed from their original storage location (i.e. storage freezers) for longer than 24 hours, and thus are to be returned by the Laboratory Services Chemist within that time period. The Chemist enters the date and time the samples were returned, and initials the Sample Retrieval Log.

10. The Laboratory Services Chemist maintains the records of the Sample Retrieval Log in a Sample Retrieval binder in the Laboratory Services Group area and must ensure that the LIMS is updated if the storage location is revised.

Storage of Sample Extracts

1. Prior to instrumental analysis, sample extracts are stored in Lab 1, in the refrigerator freezer, according to procedures described in SLA-008, "Preparing Extracts for Instrumental Analysis".

2. The long-term storage of sample extracts is the responsibility of the Instrumental Analysis group and is described in SIN-014, "Microvial Verification and Storage"

Storage of Backup Portion of Sample Extracts

1. Sample extracts, which require splitting before completion of the workup in order to preserve a backup portion, are stored in glass ampoules. The extract is transferred by the Extraction Laboratory Chemist to a clean ampoule, labelled with the Workgroup (WG) number, sample ID and splitting information. The top of the ampoule is covered with clean aluminium foil and placed in Lab 1 fridge freezer.

2. The ampoule ID is entered on the ampoule sealing record (FSA-020) located in the Sample Preparation Area. At the end of each day a Laboratory Services Chemist seals and stores all ampoules listed on the ampoule sealing record.

3. Prior to storage the ampoule ID and information is entered in the Ampoule Archive Record (FST-018) by the Laboratory Services Chemist and is initialled and dated. The location of the ampoules (Box number) is entered.

4. The ampoules are stored in boxes in Lab 7 by the Laboratory Services Chemist.

5. When an ampoule is removed for analysis a record of this is made in the "REMOVED" column of the Ampoule Archive Record. The Laboratory Services Chemist's initials, date and new WG Number are recorded.

6. The Ampoule Archive Records are maintained in an Ampoule Archive Record binder in Lab 7.

Axys Analytical Services Ltd
Standard Operating Procedure

Table 1
Summary of Storage Facilities

Storage Facility	Location	Sample Type	Comments
Walk-in Cooler (WIC1)	Storage Area on East side of South Building	Oils Wet pulp Aqueous samples XAD columns, Resin Column filters Wet and dry Ash Proofs Freeze Dried Solids	Analysis completed.
Walk-in Cooler (WIC2)	Next to Sample Receiving in West Building	Oils Wet pulp Aqueous samples XAD columns, Resin Column filters Wet and dry Ash Freeze Dried Solids	Analysis pending.
Walk-in Freezer (WIF1)	Storage Area on East side of South Building	Archived Solid and Tissue samples	Analysis completed.
Walk-in Freezer (WIF2)	Storage Area on East side of South Building	Extracts – microvials[1]	Analysis completed. In the case of back-up ampoules the analysis may still be in progress.
Walk-in Freezer (WIF3)	Storage Area on East side of South Building	Fish Study Samples	Analysis pending or completed
Walk-in Freezer (WIF4)	Next to Sample Receiving in West Building	New Samples - Solids, Tissues, Ambient Air PUF/Filters, Extracts [1, 2]	Analysis pending
Sample Storage Cupboard	Sample Receiving	Ambient Temperature Storage, All Samples for DX/F Precursors, Dry Pulps	Analysis pending

[1] Extracts that are to be analysed only for brominated dioxin/furans and brominated diphenylethers are stored 4°C, not frozen.

[2] Extracts placed in WIF4 must be double sealed in unbreakable, leak proof and airtight containers. The secondary outer container must be sufficient to prevent escape of any liquid or vapour should the primary glass container fail. Extracts that are received "injection ready" for instrumental analysis are released directly into the custody of the Instrument Group Manager or designee and stored in Laboratory 1 refrigerator/freezer.

Axys Analytical Services Ltd
Standard Operating Procedure

Title: Sample Control Procedure
Area: Laboratory Procedures

Storage Facility	Location	Sample Type	Comments
Outside Chest Freezer	Storage Area on East side of South Building	Tissue samples ready for disposal	Only those samples which require freezing.
Refrigerator	Lab 1	Solids, tissues, XAD Resin, filters, Wet pulp	Temporary storage prior to subsampling/after subsampling.
Refrigerator Freezer	Lab 1	Sample extracts [1]	Pending Instrumental Analysis
Refrigerator	Lab 5	Ampoules to be sealed	
Refrigerator Freezer	Lab 5	Working Standard Solutions	
Small Refrigerator	Lab 5	Brominated Standards	
Shelves	Lab 7	Sealed ampoules of back-up extract	Extracts sealed under nitrogen in the ampoules and stored in the dark.
Cupboards	Lab 7	Standard Solutions	Ambient Storage -Locked
Refrigerator	Lab 7	Chemical Standards	Cool Storage - Locked
Refrigerator Freezer	Lab 7	Chemical Standards	Frozen Storage - Locked
Small Fridge	Instrument Lab	Brominated microvials	Temporary storage until data approved
Standards Fridge	Instrument Lab	Instrument standards	
LCMS Fridge	Instrument Lab	PFOS samples	
Freezer	Instrument Lab	Sample extracts	Temporary storage until data approved.

References

FSA-017 Sample Retrieval Log Sample Control/17
FSA-020 Ampoule Sealing Record Sample Control /20
FST-018 Ampoule Archive Record Standard/18

Approval: _____

Axys Analytical Services Ltd
Standard Operating Procedure

Title: Sample Control Procedure
Area: Laboratory Procedures